

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

Kingdom of Belgium

Ministry of Economic Affairs

Patent Number: 852,751

International Classification: B05C

Patent Application Date: September 22, 1977

Patent Assignee: E.I. du Pont de Nemours and Company

Priorities: Two US patent applications dated March 23, 1976, number 669,601 and September 28, 1976, number 727,526, both under the names: W.D. Feathers and C. L. Long

Title: Method and Equipment for the Coatings of Films

Method and Equipment for the Coatings of Films

The invention concerns the application of liquid coating layers over a band or belt in motion. More particularly, the invention concerns a method and improved coating equipment used to apply one or several layers of high quality photosensitive compositions.

U.S. Patent number 3,508,947 describes the evolution of various coating techniques in the photographic industry and the problems encountered with them. In order to attempt to resolve these problems and more particularly to introduce higher speed coating processes, U.S. Patent number 3,632,374 suggests the use of certain coating techniques for the manufacture of photographic film. Screen or curtain coating consists of forming a liquid layer under carefully controlled conditions without imparting the coating a significant flow rate and mainly allowing it to freefall until it reaches the material to be coated. The freefall makes it possible for the liquid layer to achieve a high enough rate of speed to penetrate the layer of air surrounding the band or belt. This curtain coating removes many of the drawbacks exhibited by prior art coatings such as the strip coatings and allows for greater coating rates; however, the liquid curtain that falls is subject to disturbances due to air currents. If one were to decrease the length of the curtain drop, the air current problems are reduced to a minimum, but this reduction is not always possible to achieve since it is necessary that the drop be sufficient to give the liquid layers sufficient kinetic energy to be able to penetrate the air barrier surrounding the surface of the band if high quality coatings are desired. A solution that has been proposed and which has not had complete success consists of using deflectors behind the curtain to reduce to a minimum the effect of errant air currents.

Another solution that prevents the drawbacks of the curtain instability is described in U.S. Patent number 3,749,053. This solution consists of using a cascade coating apparatus placed very close to the band as well as a vacuum chamber to collect multiple photographic layers over a band moving horizontally. This solution exhibits a small difficulty since it needs to enlarge the opening in the vacuum box by using a spring hinge to allow the passing of the band connections.

An improvement of this solution was described starting on page 203 of the work titled, "Coating Equipment and Processes," by George L. Booth, 1970 edition, Lockwood Publishing Company, Inc. In it is proposed to subject the liquid layer to a constant acceleration along a flat gliding or sliding surface before the freefall. However, this method exhibits the drawback of subjecting the layer to excessive acceleration and to an abrupt change of direction when it goes to the freefall stage, and this results in thickness irregularities of the coating among other drawbacks.

Among the preferred embodiments, the invention proposes an improved process for the application of distinct layers over a support layer in motion. Concerning its more general aspect, the method of the invention consists of applying to a support surface at least one layer of a liquid coating composition and forcing the support surface through a coating area and at least one layer of the liquid coating composition is deposited along a gliding surface so that it can finally achieve freefall over the support surface and accelerates the layer by gravity continually increasing over the gliding surface until it flows in an essentially vertical direction.

The equipment according to the invention preferably uses a sliding surface exhibiting an increasing slope in order to ensure the controlled desired acceleration, that is, an ever increasing acceleration. By regulating the acceleration in this manner until the point of freefall and arranging it so the acceleration up to the point of final impact is produced under the conditions of regulated or controlled acceleration there is less risk of interruptions of the layer occurring than under purely freefall conditions.

The larger spacing relative to the thickness of the connectors makes it possible for them to pass through freely while only minimally disturbing the coating operation. The drawback exhibited by the instability of the curtain due to air currents is diminished by the extremely short fall. The controlled acceleration of the liquid until achieving a release rate that combined with the increase in rate while freefalling is sufficient to penetrate the air barrier over the surface of the substrate also reduces the need for additional vacuum systems or deflectors when the coatings are being carried out at high speeds, and this results in a simple process to operate.

The description and the figures provided below are meant as non-limiting examples to illustrate how the invention can be implemented. Any details or characteristics drawn from such diagrams or text are understood to be part of the invention.

Figure 1 is a vertical view of an embodiment of the coating apparatus to implement the process of the invention.

Figure 2 is a vertical view of another embodiment of a coating equipment according to the invention showing the passage of a film connection through the coating area.

Figure 3 is a partial view of a variation of the lip support of the film used in the embodiment illustrated in figure 1.

Figure 1 shows the preferred embodiment comprising all the necessary elements to form the multiple superimposed layers, their controlled acceleration at the desired rate and their deposition over a moving band in distinct layers. More precisely, a support surface or band 11, able to accept a two layer coating comprising a layer composed of a silver halide emulsion 24 and an anti-abrasive layer of the same type as those described in the examples, passing over a support roller 12 placed under the coating machine of the invention.

The coating equipment shown in figure 1 resembles in part the prior art coating machines known as flowing or gliding hoppers that have been used for weather striping. The body of the hopper comprises four sections. Sections 15 and 16 define a first feed source 17 and a first reservoir 19 through which a liquid is pumped by means of pump 26 and pipe 27. The upper part of section 15 is flat and polished and forms an inclined gliding surface 20. In a similar fashion, sections 14

and 15 define a second source 18 and a second reservoir 30 through which the liquid can travel pumped by pump 29 and pipe 28 over the upper surface of section 14. The upper surface of section 14 is on the level and on the plane located slightly lower than the plane of the sliding surface 20. The upper surface of section 14 is also flat and polished and forms a second gliding surface, the angle with the horizontal is practically identical to that of the gliding surface 20. The distance where the plane of the second gliding surface 21 is withdrawn under the plane of the first gliding surface 20 and it is at least equal to the desired thickness of the liquid layer pumped through source 18 and flow over the gliding surface 21.

The equipment for the production of multiple layers as described up to now is the traditional one. According to the invention, section 13 defines an upper surface at the convex curve 22 and it is attached to the end of section 14 on the side opposite to that which is connected to section 15. According to a preferred embodiment, the gliding surface at convex curve 22 starts from the end of the flat gliding surface 21 and ends on it slower edge 60 at a point tangent to the vertical located at a distance "d" of band 11. Preferably the distance "d" can be varied to permit the free passage of a band connection, a distance that can be as little as 0.8 mm or as large as 19 mm or there about or that could be approximately 25 mm when the band rate is high. Preferably, distance "d" must be maintained as small as possible in order to prevent the harmful effects due to air turbulence over the liquid layer.

The selection of the particular shape of the convex curve of the sliding surface 22 along the direction of the flow may vary depending on the rheological properties of the coating solution, the desired weight of the coating and the speed/rate of band 11. It has been found that a surface close in shape to a cone that is having a mostly parabolic shape along the direction of the flow of the type defined by the equation $y = -x^2/k$, where x is the abscissa and y the ordinate of a diagram defined by this equation produces good results in the sense that the variation of acceleration of the coating solution during its displacement along the sliding surface is constant. However, other convex shapes are acceptable also and could be useful to satisfy the parameters indicated. Transversally to the direction of the flow, the surface defines a straight line. In every case, it is preferable that the proportional increase of the speed imparted to the coating solution under controlled acceleration determined by the gliding surface relative to the proportional increase of speed imparted to the coating solution during freefall, is increased as much as possible. Proportional increases in rate under controlled conditions can best be achieved with liquids of lower viscosities.

The pouring edge 60 is represented by a square section in order to provide a starting point of the liquid that is well defined and prevents an excessive melting of the edge and consequently the formation of striations in the coating that has been deposited. The pouring edge can also have a rounded section as shown in figure 3 or a knife blade section as shown on figure 2. The essential condition is that the pouring edge does not hinder the flow of the liquid that is preferably at the desired impact rate when it reaches this point.

Even though it has been stated that the pouring end of the gliding surface is preferably oriented vertically that is $\alpha = 90^\circ$, the angle α may vary from approximately 80 to 120° depending on the impact rate desired, the parameters of the liquid, etc. Angles of less than 90° are not the preferred ones since the liquids have a tendency to prematurely leave the sliding surface. Equally, angles that are significantly greater than 90° are not preferred either since they tend to bring about an

abrupt change in the acceleration of the liquid as they leave edge 60. This is contrary to the object of the invention, which is to provide a transitional flow area so that the flowing layers pass to freefall with a minimum acceleration change. A surface that is essentially vertical is preferable, that is $80^\circ < \alpha < 120^\circ$.

Figure 2 shows a variation of the invention. In this variation, an arch is used to define the sliding surface 32 in section 33 of the coating apparatus. Belt or band 11 in figure 1 is replaced by two bands 44 and 42 to illustrate how a connection passes by or through the coating site. The equipment of figure 2, as it is shown, is designed to apply only one layer and the body of the coating machine is made of two sections 33 and 46 that define a feed source 38 and a reservoir 39. Pump 36 brings the liquid to the upper part of section 33 of the body through pipe 37. The upper part of section 33 of the body comprises a flat sliding/gliding section on an inclined plane 31, which extends up to point A beyond which it curves, thus forming a portion of the circle with radius R that ends in point B tangent to the vertical located at distance "d" of moving band 42. Band 42 passes over guiding roller 12. Figure 2 shows a connection in the band that passes by the coating site under the pouring edge 62. The back end 40 of band 42 is applied over a part of the front end 43 of band 44. Over the coating part of the two bands 44 and 42 is placed a piece of adhesive tape 41, which typically extends over the entire length of the band. The slope of the gliding surface may be varied as stated above and shown in figure 1, and also the distance "d" can be adjusted to adapt to the different coating compositions at different belt rates and at the desired impact rates.

It is understood that according to the invention other known styles of hoppers may also be used such as an extrusion hopper to produce superimposed distinct layers before accelerating them over a convex gliding surface.

The vertical extremity of the convex gliding surface is represented over the axis of the guiding roller 12 on figure 1 and it is off center on figure 2, but it is understood that this position may vary and that it may be located at any point on the band where it is preferably placed horizontally or close to it. However, it is preferred that the band is supported by a roller on the coating site to reduce to a minimum the disturbances due to the flotation. It is preferred that the band is placed horizontally on the point of impact of the liquid layer; however, this can also be achieved anywhere on the curved part of band 11 that performs the tour of the roller 12 (figure 1) close to positions of 9 o'clock and 12 o'clock. Equally, it is not necessary that the band be held at the point of impact but a support is recommended.

Returning to figure 1 to describe the process of the invention. Band 11 passes over roller 12 and it is put in motion in the direction that the arrows indicate at the desired coating speed by an appropriate machine (not shown). A liquid that may be an emulsion of silver halide type described in the examples or any other similar solution used in photography or in other coating techniques is pumped from a container not shown by pump 29 and pipe 28 in order to fill reservoir 30 that serves a pressure balance. The liquid goes through source 18 to arrive at the lower gliding surface 21. Preferably source 18 extends all along gliding surface 21 when reservoir 30 is full, the liquid rises in source 18 and spills over surface 21 where it starts to flow downwards due to gravity. The flow rate of the liquid is such that it forms a uniform layer 24 covering the entire length of the gliding surface 21 that may be positioned to create a slope of approximately 23° . In fact, the slope of the gliding surface can be adjusted between

approximately 0° (with an extrusion hopper) and approximately 45° (with an extrusion hopper or gliding hopper) depending on the compositions used, etc. The slope is selected in a manner that makes it possible to form liquid layers and allows them to descend to the convex area on the gliding surface.

From a second reservoir not shown, pump 26 brings a second layer that can be an anti-abrasive layer comprising a 6% gelatinous or gel like aqueous solution, antistatic agents and hardeners used in the art. This second liquid is directed by pipe 27 in order to fill reservoir 19 then through slot 17 in order to form a uniformly flowing layer 23 over the upper gliding surface 20. When layer 23 flows downward, it finally achieves the exit from slot 18 and continues its descending flow over layer 24, the two layers now flow in distinct superimposed layers.

When the layers reach the end of the inclined flat surface 21 and reach convex surface 22, their speed increases by gravity proportionally to the variation of the curvature of the gliding surface. When the layers reach the end of the convex gliding surface 22, they accelerate due to the appropriate curvature selection at a rate that combined with the increase in speed over the short freefall is sufficient to penetrate the air barrier over the band and is deposited over the belt in motion 11. The two layers 23 and 24 occupy the space between edge 60 of the surface 22 and belt 11 by freefalling from a height "d" in a continuous current 25. Given that the liquid layers pass in freefall at a significantly higher rate than is recommended by the curtain method, it is no longer necessary to have freefall over great distances to achieve a sufficient impact rate to overcome the problems posed by the air currents and significantly shorter drops may be used. Distance "d" may then be a minimum.

When a connection reaches the coating site as shown in figure 2, the flow of the coating liquid is not disturbed as long as the distance "d" is such that the connection can pass without breaking liquid current 25.

The following examples illustrate the invention without limiting it.

EXAMPLE 1

Equipment such as shown in figure 1 is used to apply to a support in motion a gel-silver halide emulsion prepared in the traditional manner comprised of a silver halide in suspension in an aqueous gel solution. The support is composed of an ethylene terephthalate sheet of the type used in the manufacture of photographic film essentially prepared in the manner described in U.S. Patent number 2,779,784. The thickness of the support is 0.18 mm.

In this case only one layer is applied so that pump 26, does not operate during this experiment and the liquid is brought to the gliding surface by pump 29 and slit/aperture 18. The convex gliding surface is parabolic and follows equation $y = -x^2$ and starts from a point tangential to the gliding surface that makes a 23° angle with the horizontal plane. The total length in the direction of the flow of the liquid is 12.7 cm. The machine is able to apply a layer 11.75 cm long over the moving belt. The viscosity of the emulsion is maintained at 9.7 centipoises at 38°C and the liquid is brought to the machine at a rate of 720 ml/min. Distance "d" may range between 3.2 and 12.7 mm. Satisfactory continuous coatings are obtained when the band moves at 76.2 cm/sec. The calculated weight of the dry coating is 149 mg/dm².

EXAMPLE 2

Using the equipment described in example 1, but this time with a generally parabolic convex gliding surface of approximately 17.8 cm long, only one 10.2 m layer is deposited comprised of a sensitized silver halide emulsion containing silver halides in suspension in an aqueous gel solution over a moving belt of the type described in example 1 at a rate of 223.5 cm/sec. The rate of the solution is 720 ml/min and its viscosity measured at 35°C is 11.4 centipoises. The dropping distance "d" ranges from 4.8 to 6.3 mm approximately. The dry weight of the coating is 75.5 mg/dm². All coatings were judged to be satisfactory.

The invention has been described by the embodiments considered most advantageous, but these are not meant to be limiting. For example, hopper or pourers may be used as the sources of liquid layers as laminar flows. We can also use a combination of dies and gliding hoppers to produce multiple layers. Also edge guides may be used for coating over the gliding surface and across the fall distance "d" as indicated in U.S. Patent 3,632,374 to maintain the total length integrity of the coating.

CLAIMS

1. Method for the application of at least one layer of a liquid coating composition over a support surface consisting of passing this surface through a coating area and making at least one layer of this liquid composition flow over a gliding surface in a manner that it ends up attaining a freefall over the support surface, characterized in that the layer accelerates by gravity continually increasing over the gliding surface until it flows in a generally vertical direction.
2. Method according to claim 1, characterized in that said increase is constant.
3. Method according to claims 1 and 2, characterized in that the gliding surface is defined within the direction of the flow by a cone shaped section.
4. Coating apparatus for coating at least one layer of a coating composition over a support surface having an incline surface, a source of the liquid coating composition, the means to form a layer of said composition over said surface and the means to make this support surface under the gliding surface, characterized in that the gliding surface has an edge and an ever increasing slope from the point of formation of the layer to the edge.
5. Method according to claim 4, characterized in that the gliding surface defines a conical shape in the direction of the flow.

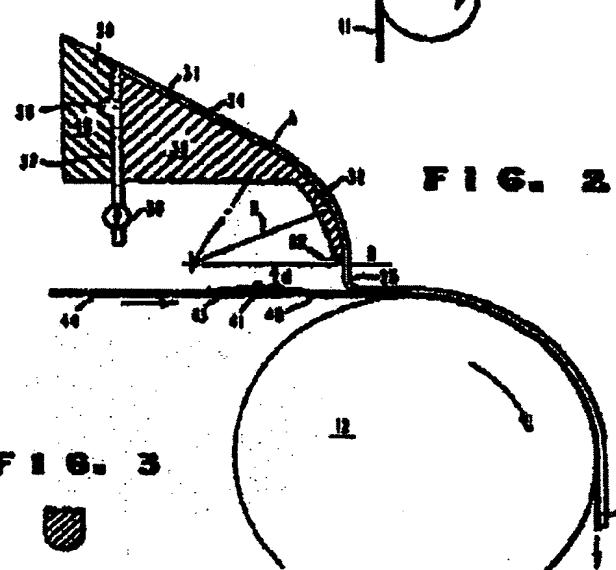
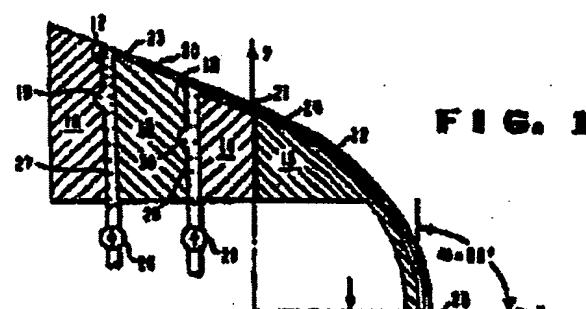


FIG. 3

